NISTIR 89-3913

INTERLABORATORY COMPARISON OF THE GUARDED HORIZONTAL PIPE-TEST APPARATUS

Precision of ASTM Standard Test Method C-335 Applied to Mineral-Fiber Pipe Insulation

David R. Smith

NISTIR 89-3913

INTERLABORATORY COMPARISON OF THE GUARDED HORIZONTAL PIPE-TEST APPARATUS

Precision of ASTM Standard Test Method C-335 Applied to Mineral-Fiber Pipe Insulation

David R. Smith

Chemical Engineering Science Division
Center for Chemical Engineering
National Institute of Standards and Technology
Boulder, Colorado 80303-3328

April 1989

U.S. DEPARTMENT OF COMMERCE, Robert A. Mosbacher, Secretary

NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY, Raymond G. Kammer, Acting Director

CONTENTS

LIST OF TABLES

LIST OF FIGURES

INTERLABORATORY COMPARISON OF
THE GUARDED HORIZONTAL PIPE-TEST APPARATUS

Precision of
ASTM Standard Test Method C 335
Applied to Mineral-Fiber Pipe Insulation

David R. Smith

NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY
BOULDER, COLORADO 80303-3328

Apparent thermal conductivity of refractory pipe insulation from the same production lot was measured by seven different laboratories. The purpose of this intercomparison was to assess the precision and bias of the ASTM Test for Measurement of Steady-State Heat-Transfer Properties of Horizontal Pipe Insulation (C 335). Four laboratories submitted conductivity measurements for conditioned specimens. The test results for thermal conductivity of the four conditioned specimens include the temperature range from 318 to 652 K and illustrate the interlaboratory reproducibility as well as the temperature dependence of the conductivity. The standard deviation of the data from each participating laboratory was measured by their deviation from a polynomial function fitted to all data. For the 16 test results from the four laboratories that conditioned their specimens, the standard deviation was 4.3 percent. For all test results from all seven participants, the standard deviation was 5 percent. This value is offered as the estimated precision of the horizontal pipe-test method. The accuracy of this pipe-test method cannot be estimated from the data obtained in this intercomparison.

Key words: accuracy; apparent thermal conductivity; bias; guarded hot plate; high temperature; horizontal pipe; insulation; interlaboratory comparison; mineral fiber; precision; standard test method.

1

1. INTRODUCTION

A firm principle of measurement science is that a number for the magnitude of a physical property such as thermal conductivity conveys little information unless the precision and accuracy of the numerical value are also given. The wise user seeks to know the precision and accuracy of the value in order to understand the confidence that he or she legitimately can give to the information. Manufacturers of products such as thermal insulation, whose fitness for intended use depends on specific physical properties, are ethically and legally obligated to claim no more for the performance of their products than can be objectively demonstrated by accepted standard test methods. Thus manufacturers also need to know the precision and accuracy of numbers they want to use to represent the performance of their products. Then they can honestly advertise all the benefits that their products are able to deliver to the consumer, without subjecting themselves to legal problems arising from misrepresentations.

Considerable progress has been made during the last twenty years in improving equipment and techniques described by standard ASTM test methods for measuring heat transfer in insulations, especially those used in the building industries, at temperatures near ambient. However there has been only limited knowledge available of the imprecision and bias of the ASTM test methods when they are extended to higher temperatures. During the last few years a concerted effort has been under way to evaluate the imprecision and bias of the various ASTM test methods for measuring heat transport through thermal insulations. The methods involving measurements at temperatures several hundred kelvins above room temperature are currently of particular interest.

Of the several instruments used to measure apparent thermal conductivity of insulation materials, two are important because they are independent of calibration materials and may be considered to embody "absolute" methods. These two instruments are the guarded hot plate (GHP), for specimens in the shape of flat slabs, and the horizontal pipe-test apparatus, for specimens in the shape of cylindrical shells. The laboratories of the U.S. National Institute of Standards and Technology, at Gaithersburg, Maryland (NIST-G), and at Boulder, Colorado (NIST-B), have played an active role in helping to improve equipment, focussing on guarded hot plates [1-4]. Apparent thermal conductivity of two matched specimens can be measured near room temperature with an accuracy of about 2 percent [5] in a guarded hot plate complying with ASTM Standard Test Method C 177 for "Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate Apparatus" [6].

For measuring thermal conductivity of cylindrical pipe insulation the relevant ASTM test method is C 335, "Steady-State Heat Transfer Properties of Horizontal Pipe Insulation" [7], commonly referred to as the "horizontal pipe-test" (HPT) method. The imprecision and bias of this test method have not yet been established, and estimating these is the issue addressed by this report.

2

As a consequence of the incomplete knowledge of the imprecision and bias of the HPT method over the higher range of temperatures typically used in practice, seven interested members of ASTM Subcommittee C-16.30 (Thermal Measurements) organized an interlaboratory comparison on this method in the fall of 1987. The objective was to measure specimens of homogeneous pipe insulation at mean temperatures between ambient and about 673 K (400°C). Members of the task group wanted to move quickly to learn the present state of the quality of measurements with this method. To save time and to reduce damage to the specimen, they decided not to pass around the same specimen of pipe insulation for measurement, but rather to measure concurrently specimens chosen from the same production lot. The deadline for completion of the measurements was chosen to permit results to be presented in the spring of 1988 at the ASTM C-16.30 meeting in Atlanta, Georgia.

2. COMPARISON OF THE GUARDED-HOT-PLATE AND PIPE-TEST METHODS

The recent experience in determining the imprecision and the bias of the guarded hot plate is relevant to efforts to determine them for the HPT method. The similarities and the differences between the two types of apparatus help us to understand the possible causes of imprecision and bias in both instruments.

a. The Guarded Hot Plate

To assess the imprecision and bias of high-temperature GHPs, an interlaboratory comparison of measurements of apparent thermal conductivity was held in 1987 [8]. The study used GHPs designed for high temperature to measure fibrous alumina-silica and calcium silicate insulation boards as test specimens. This intercomparison showed that, for mean specimen temperatures ranging from 313 to 773 K, considerably larger uncertainties exist in measurements made with such GHPs than for room-temperature measurements. For the fibrous alumina-silica specimens the greatest deviations of the participants' data from a least-squares fitted curve were +20 and -12 percent, and the standard deviation was 7.4 percent. For calcium silicate specimens the greatest deviations of the participants' data from the fitted curve were +13 and -22 percent, and the standard deviation was 8.0 percent. Thus for the GHP considered as a standard ASTM instrument for measuring thermal conductivity at high temperatures, the standard deviation of its data from a fitted descriptor curve is almost 8 percent.

This was particularly surprising, because the GHP method is considered to be an "absolute" test method for determining thermal conductivity of insulations, because no calibration specimen is used with this apparatus. The conductivity of the specimens is simply calculated from several quantities obtained during the experiment and from the geometry of the specimen.

The thermal conductivity of a specimen can be defined through Fourier's law as the ratio of heat flux, q, to the magnitude [dT/dX] of the thermal gradient (where square brackets denote the absolute value):

$$k = q/[dT/dX].$$ (1)

The heat flux q is defined in terms of two physical quantities measured for the metered area of the source of heat. These quantities are the rate of transport Q of thermal energy from the heater through the adjacent area of specimen, and the corresponding cross-sectional area A through which the energy is conducted. The ratio of these, Q/A, defines the heat flux q through the metered area. Two other quantities obtained during the course of the experiment are the temperature difference ΔT along the direction of heat flow, together with the corresponding thickness of specimen ΔX. However the ratio of these, $\Delta T/\Delta X$, only approximates the thermal gradient dT/dX required by Fourier's law. In practice, k is defined by a slight modification of relation (1), obtained by a single integration:

$$\int_1^2 k(T)dT = (Q/A)\cdot\Delta X,$$ (2)

where ΔX is the spatial separation between the surfaces at temperatures T_1 and T_2 bounding the specimen. Of course this relation (2) only implicitly specifies k.

The integral on the left side of this relation may be replaced by an equivalent quantity, $k_{mean}\cdot(T_2-T_1)$. Alternatively, the GHP is often operated with temperature differences small enough that k(T) may be estimated by a constant, k_{mean}, over the applicable temperature interval. In either case, relation (2) takes a form which then explicitly defines the experimental thermal conductivity:

$$k_{mean} = (Q/A)\cdot\Delta X/(T_2-T_1).$$ (3)

The correctness of the GHP method is determined in practice by how nearly the apparatus approximates, within the metered area of the specimen, uniform and unidirectional flow of heat through an infinitely broad slab of material which has uniformly heated, par-allel, planar surfaces and which is homogeneous along directions

4

parallel to the surfaces. The boundary temperatures at the surfaces of the specimens are maintained by heated (or cooled) plates whose temperatures are carefully controlled. If the specimens have a constant thermal conductivity (independent of temperature), the thermal gradient dT/dX within the specimens is independent of position along the direction of heat flow in the test area, and then is exactly given by $\Delta T/\Delta X$. Any flow of heat into or out of the perimeter of the measured central area, or in other words, heat flow that has a component along the normal to the axis of symmetry within the metered area, leads to an erroneous value of thermal conductivity.

The existence of large differences among measurements carried out by different laboratories using the GHP method [8] indicates the possibility of uncompensated biases in measurement of the heat flux or of the thermal gradient. One possible source of bias is faulty thermal grounding, or tempering, of the leads of the thermocouples which measure the temperature difference from which the thermal gradient is calculated. Most GHP devices are 20 to 25 cm in diameter, with a consequent tempering path length of only 10 to 12 cm for the thermocouples measuring the thermal gradient at the center of the specimen. One exception is the NIST-G 1-meter GHP [1,3], whose diameter leads to a greater length of tempering of the thermocouples used to measure the temperatures at the surfaces of the specimens. Other exceptions are the two (low-temperature and high-temperature) GHPs at NIST-B [2,4], which use extra tempering lugs to increase the effective length of thermal grounding of the thermocouple leads as they exit the GHP stack.

b.   The Horizontal Pipe-Test Apparatus

Using the same criteria applied above to the GHP method, the ASTM C-335 HPT method for horizontal pipe insulation [7] could also be considered to be an absolute test method, although it is not at present so regarded. Like the guarded-hot-plate method, the C-335 HPT method is designed to simulate an ideal geometry of unidirectional heat flow. As with the GHP method, no calibration specimen is necessary with the HPT apparatus, and the conductivity is also calculated as the ratio of heat flux q to the (radial) thermal gradient, dT/dr, both being obtained in the course of the experiment.

One important operational difference between the two methods is that the GHP method uses relatively small temperature differences in measuring thermal conductivity, and uses an auxiliary heater to vary the mean temperature of the specimen independently of the magnitude of the temperature difference within it. In contrast, the HPT method relies on ambient (although controlled within 1°C) temperature to maintain the cold-side temperature of the specimen. It is not required that the HPT apparatus have an auxiliary heater with which to vary the mean temperature. It is permissible to vary the mean temperature by increasing the hot-side temperature, with a large difference of temperature within the specimen.

5

With the HPT method, the cylindrical geometry of the physical apparatus used in this test method also complicates matters slightly. The heat flux q is the ratio of the rate heat of heat flow Q to a cylindrical area $2\pi rL$ located at the radial position r measured from the central line heater and occupying an interval L along the axis of symmetry. The position r is that at which the gradient, dT/dr, is evaluated. For this cylindrical geometry, Fourier's law takes the form,

$$Q/2\pi rL = k \cdot dT/dr. \qquad (4)$$

The presence of the r factor in the left side makes the temperature gradient dT/dr a function of position r even if k is constant. This expression (4) must be integrated with respect to radial position to obtain a result applicable to the finite differences in T and r used in an actual experiment. This is easily done, and results in the expression

$$(Q/2\pi L)\ln(r_2/r_1) = \int_1^2 k(T)dT, \qquad (5)$$

where r_2 and r_1 are the outer and inner radii of the cylindrical insulation specimen.

The factors r_1, r_2, and L are constants during a given experiment. The <u>integral</u> of the thermal conductivity function between the temperatures T_1 and T_2 of the bounding surfaces is the conductivity-related quantity proportional to the heater power Q. It is the integral of thermal conductivity which is obtained from the conditions of the experiment (Q, r_1, r_2 and L), rather than the thermal conductivity alone. This integral of k(T) over T is mathematically equivalent to a product of conductivity and temperature, and may be used to define a "mean" conductivity by

$$k_{mean} = \int_1^2 k(T)dt/(T_2 - T_1), \qquad (6)$$

which is generally not the same as $k(T_{mean})$ [11].

The correctness of the HPT method is determined by how nearly the HPT apparatus approximates purely radial flow of heat, from a uniform source of heat at the center of symmetry, through an infinitely long cylinder of material which has concentric cylindrical surfaces and which is homogeneous along directions parallel to the surfaces. The boundary temperature at the inner surface of the specimen is maintained by a cylindrical heated pipe, whose temperature is carefully controlled. However, in contrast to the case of the GHP, the temperature at the outer (cooler) surface of the insulation is not maintained by a temperature-controlled surface, but rather by the ambient, laboratory temperature. Even though the laboratory temperature may be controlled, the temperature of the specimen surface is not as tightly controlled as it would be by a cooled surface in thermal contact with it. Also, because of the cylindrical geometry, which causes the heat flux lines to diverge from the linear center of symmetry, the temperature gradient within the specimen must always be a function of position from the central heater, in contrast to the situation with the GHP method. This is true even if the conductivity is independent of position along the direction of heat flow.

Any flow of heat along the direction parallel to the cylindrical symmetry axis, into or out of the boundaries of the measurement length, or in other words, heat flow that has a component parallel to the axis of symmetry, would lead to an erroneous value of thermal conductivity. The temperatures defining the thermal gradient are measured by thermocouple leads introduced along a cylindrical isothermal surface. A greater length of thermal grounding of the leads, typically 30 to 45 cm, is possible in comparison to the length available with the disk-shaped heater plates in the GHP. This is so because of the commonly encountered combined length (60 to 90 cm) of the test and guard regions within the cylindrical HPT apparatus.

Only the GHP method has found wide acceptance in the U.S. as the prime method for measuring thermal conductivity of insulating materials. One major factor is that the GHP method uses specimens in the shape of flat slabs, which is the most common geometry for insulation materials. Although each GHP apparatus has a definite limit on the maximum thickness of specimen whose conductivity it can measure accurately, specimens of any thickness less than that maximum may be measured. The cylindrical geometry of the HPT method is on the other hand somewhat restrictive because a given apparatus can measure only specimens of radius matching that of the cylindrical inner surface, even though any size of outer surface may be used. Manufacture of homogeneous insulation specimens of cylindrical shape is somewhat more difficult than that of flat-slab specimens.

To date no systematic comparison has been carried out to link results on identical materials measured with both the guarded hot plate and the HPT methods. Such a comparison would be a valuable test of the accuracy and bias of both methods, in light of the existing uncertainty in measurements with high-temperature GHPs.

7

One study by Kimball [9] of a large HPT apparatus compared calculated steady-state thermal conductance (rate of heat flow per unit area, per kelvin) with experimental measurements, for nominal 3-inch polyurethane foam insulation, used on 18-inch pipelines in the Arctic. He reported measuring conductance through the insulation with an exterior temperature of -56.7ºC (-70ºF) and an interior (pipe) temperature of +71ºC (+160ºF). The experimental thermal conductance was 6 percent higher than the theoretical. This discrepancy was explained by heat leak from the interior along the central boundary joining the two abutting sections making up the complete specimen. A measurement of the conductance at 7.2ºC (45ºF) under transient conditions agreed within 4 percent with the corresponding calculated value.

The only previous interlaboratory comparison of the test method for horizontal pipe insulation took place over a period of more than seven years and was reported by Hollingsworth [10] in 1977. Ten laboratories took part in this comparison, which covered a range of mean specimen temperatures from about 323 to 423 K; the highest pipe-heater temperature used was about 533 K (260ºC). Hollingsworth concluded that the test error was approximately 3 percent. However, both the HPT method and the insulations tested with it are frequently used at mean temperatures much higher than the upper limit of 423 K of the 1977 intercomparison.

3. SCOPE

This report analyzes apparent thermal conductivity of high-temperature pipe insulation measured by seven testing laboratories using guarded horizontal pipe-test apparatuses. Their instruments comply with ASTM test method C 335 for "Steady-State Heat Transfer Properties of Horizontal Pipe Insulation." The specimens measured were homogeneous mineral-fiber insulation designed for use at temperatures up to 600 K or more. Four of the testing laboratories submitted data for "conditioned" specimens, over the range of mean temperatures from 318 to 652 K. The results presented here apply specifically only to homogeneous specimens, to measurements within the cited range of temperatures, and to guarded, horizontal pipe-test apparatus. Extension of these results to inhomogeneous specimens, to calibrated rather than guarded apparatus, to vertically oriented apparatus, or to temperatures outside the cited range, is inappropriate.

Only one nominal density of pipe insulation was measured. Furthermore, visual inspection shows that the pipe insulation material has relatively large pores, with dimensions of up to tenths of a millimeter. Only for an insulation material for which all pores have dimensions of less than micrometers ("microporous") would pressure dependence be a factor in its thermal conductivity. This implies that intercomparisons of the thermal conductivity data reported here do not have to be corrected for the elevation above sea level of the different participating laboratories.

8

4. PARTICIPANTS AND APPARATUSES

The chairman of the task group, who took part in the inter-comparison, carefully selected some specimens from one supplier and distributed them to the other six participating laboratories. After the thermal conductivity was measured the seven participants submitted their data directly to me for analysis.

The names of the seven laboratories participating in the intercomparison are given here in alphabetic order to identify the sources of the data presented. Listing these names does not in any way imply endorsement of the organizations, or of their products or services, either by NIST in particular, or by the U.S. government. The seven participants were:

California Bureau of Home Furnishings and Thermal Insulation
CertainTeed Corporation, Blue Bell, Pennsylvania
Dynatech Scientific, Inc. (now Holometrix), Cambridge, Mass.
Fiberglas Canada, Inc., Sarnia, Ontario, CANADA
Manville Corporation, Denver, Colorado
Owens-Corning Fiberglas, Granville, Ohio
PABCO, Fruita, Colorado

Random numbers and symbols were assigned to the data sets from the participants and used throughout the figures in this report. The symbols chosen are listed in table 1, along with the characteristics of the horizontal HPT apparatus used by the participants. All apparatuses were "guarded" rather than "calibrated" HPT apparatuses, and all had nominal diameters of 3 in (actual diameters of 3.5 in, or 89 mm).

Table 1. Coding symbols used in figures to distinguish the seven laboratories participating in the interlaboratory comparison, and sizes of their nominally 3-inch pipe-test apparatus.

Lab Code	Lab Symbol	Metered Length (cm)	(in)	Guard Length (cm)	(in)
1	O	91.4	36	30.5	12
2	△	61.0	24	15.2	6
3	□	91.4	36	30.5	12
4	▽	61.0	24	7.6	3
5	◇	91.4	36	30.5	12
6	+	61.0	24	15.2	6
7	✕	61.0	24	10.2	4

---

5. SPECIMEN CONDITIONING

When the test results were submitted and presented at the At- lanta meeting of ASTM C 16.30 in the spring of 1988, it was real- ized from the order in which the data had been submitted that an important item had been overlooked. Five participants had measured the thermal conductivities of their specimens with mean temperature increasing from one measurement to the next. However, the speci- mens may change slightly as the temperature increases during the sequence of measurements leading to the highest temperature of measurement. For example, some binder begins to burn out of the insulation when it is heated to temperatures above about 400 K; other physical changes, such as loss of adsorbed water, followed at still higher temperatures by loss of water of crystallization, can take place to some degree, depending on the composition of the insulation material and on the highest temperature to which the specimen is subjected. Such changes, although minor in their over- all effect, still lead to variation in the physical properties of the specimen under study. Except for loss of adsorbed moisture from the surfaces of the pores, these changes are irreversible and permanent. Each increase in mean temperature alters the specimen: therefore successive measurements on the "same" specimen are actually being performed on slightly different specimens.

On the other hand, we can hope that the specimens of insula- tion material as received from the manufacturer have already been uniformly conditioned by the manufacturing process. Thus even in the presence of physical changes caused by heating to the temperatures used in this study, all specimens have identical histories during comparative measurements of conductivity up to the lowest maximum temperature employed by the group of participants. If heating the specimens causes physical changes affecting the thermal conductivity, then only specimens heated to temperatures greater than the maximum of another participant are different from that participant's specimen. Of course, the specimen heated to the group's maximum of temperature becomes unique.

Even if we accept this view, after the maximum temperature is reached the properties of a particular specimen depend on what that maximum was; no further loss of binder or of water of crystalliza- tion should take place unless the maximum temperature is later exceeded. Measurements of conductivity thereafter should be repro- ducible, but they will be different from those initially obtained in the first excursion from low temperatures to the maximum. Thus we still conclude that, if we want to know what the conductivity of the material is, we should use only the data obtained after the specimen has reached its maximum temperature.

To obtain stable and reproducible results, specimens of high- temperature pipe insulation should first be conditioned, or stabil- ized, by heating to the highest temperature of measurement when this lies more than about 80 K above room temperature. Also, the conditioning temperature should be the same for all laboratories in

10

the intercomparison. Only then are the different specimens stable and suitable for use in an interlaboratory comparison. However one other difficulty is that not all laboratories could reach the same highest temperature, and the highest temperature that all could reach was not determined before the intercomparison began. For these reasons, slight variations in the heat-transfer properties of the material were unavoidable. These variations, which are actually material variability, may appear disguised as interlaboratory variation in the data submitted by the participants.

Five laboratories had measured thermal conductivity before stabilizing their specimens at the highest temperature. These participants agreed, as time allowed, to remeasure their specimens before the C-16.30 Tucson meeting in the fall of 1988. Two laboratories were able to submit data for their repeated measurements. All data submitted by the participants in the intercomparison are presented here for review. Because of differences in conditioning of the test specimens, several different points of view will be used to critically examine the conductivity measurements in order to estimate the imprecision and the bias of the horizontal pipe-test method as presently standardized by ASTM method C 335.

6. THERMAL CONDUCTIVITY DATA

The thermal conductivity data submitted by the participants on the standardized forms provided to them were compiled into tabular files for analysis by computer. One participant submitted data directly in SI units; the remaining data, submitted in engineering units, were converted to SI units for uniformity and ease of comparison.

Table 2 contains all the test data for the pipe insulation used in this intercomparison (in the column headed K_{dat}). In particular, in table 2 the first four lines of data for laboratories 2 and 4, and all the data for laboratories 3, 5 and 7 are for non-conditioned specimens (data taken with temperature increased between measurements). Because the fourth line of data for laboratory 4 is for the maximum temperature of measurement, it is effectively a conditioning point; thus the last line for laboratory 4, and the fourth line, are for a conditioned specimen. All the data for laboratories 1 and 6, and the last four lines of data for laboratory 2 are also for conditioned specimens. The complete data set is plotted in figure 1, with seven different symbols used to distinguish the data from the seven participants. As is conventional, the points are plotted in figure 1 at a value of temperature equal to the mean of the two boundary temperatures. However the experimental values of thermal conductivity (K_{dat}) are averages of the conductivity over the complete temperature interval and are therefore to be associated with that specific interval.

7. REFERENCE EQUATIONS AND DATA ANALYSIS

It is now widely appreciated that several factors contribute to the total transfer of heat from one boundary surface to the other in most insulations. The main contribution is usually conductive, due to diffusive flow of heat driven by a temperature <u>gradient</u>, or spatial rate of change of temperature, through the solids and surrounding gas. However another important component is radiative heat transfer, for which the radiated energy flux per unit time is driven not by a spatial gradient but by the <u>difference</u> between the fourth powers of the temperatures at the bounding surfaces:

$$q_{rad} = B \cdot (T_H^4 - T_C^4), \tag{7}$$

where B is a combination of several constants. The quantity in parentheses can be algebraically factored into several terms, one of which is the difference $(T_H - T_C)$ between the boundary temperatures. Thus this relation can be cast into the form of Fourier's law of conduction involving the usual approximation $\Delta T/\Delta X$ to the temperature gradient: one then obtains an effective "radiative" conductivity. Equation (6) can be transformed into an algebraically identical form:

$$q_{rad} = [B \cdot (T_H^2 + T_C^2) \cdot (T_H + T_C) \cdot \Delta X] \, \Delta T/\Delta X, \tag{8}$$

where ΔT is the temperature difference $(T_H - T_C)$, and the quantity in square brackets is the "radiative" conductivity parameter, obtained at the cost of introducing into it a dependence on specimen thickness ΔX. An additional result of interest is that this "radiative conductivity" varies approximately as T_{mean}^3, where T_{mean} is the arithmetic mean of T_H and T_C.

Fourier's law of conduction, which relates the conductive heat transfer to the thermal gradient, only approximately models the total heat transfer within an insulation. The total transfer of heat between two different isotherms within an insulation material depends partly on the difference of the temperatures if radiative transport of heat is appreciable. This radiative heat transfer term may be added to the conductive term under the (only approximately valid) assumption that they represent non-interactive and parallel, or linearly additive, modes of heat transfer. The resultant total heat transfer, which is predominantly, but not purely, conductive, is called the "apparent" thermal conductivity to distinguish it from a true conductivity driven only by a thermal gradient. For brevity, the term "thermal conductivity" will be used in the following text and figures.

12

In the analyses below, the "thermal conductivity integral" method [11,12] was used in fitting thermal conductivity functions to experimental data. That is, a specific polynomial for the thermal conductivity as a function of temperature was assumed, and then integrated, and the polynomial function was fitted to the data by means of a least-squares fit to obtain the numerical values of the coefficients in the assumed relation. The deviations shown in the tables and in the deviation plots were also calculated by the integral method. This was done to remove any potential inconsistency caused by the presence of both large and small temperature differences in the data set. The use of large temperature differences is inherent in ASTM method C 335 for measurements of pipe insulation at mean temperatures well above room temperature.

When we fitted functional curves to the experimental conductivity data, weighted least-squares fits were used; the weighting was chosen to minimize the sum of the squares of the relative deviations, rather than the sum of squares of absolute deviations. This is considered preferable because when measuring thermal conductivity, experimentalists tend to keep the relative (percentage) errors constant.

The seven participants' complete set of data for the thermal conductivity of pipe insulation extends over the range of mean temperatures from 318 to 652 K (table 2). These data include measurements for both conditioned and nonconditioned specimens. The variation of conductivity with temperature for the complete data set can be represented by the correlation

$$k = 16.734 + 0.04180 \ T + 0.17954*10^{-6} \ T^3, \qquad (9)$$

where k is in mW/(m·K) and T is in K; this relation is plotted as the solid line in figure 1. The presence of the term in T^3 represents an expected contribution from "radiative" conductivity. The coefficients in eq(9) were obtained from a weighted least-squares fit of the data, for specimens having a mean density of 91 kg/m³. The standard deviation of density about the mean is 4.55 percent.

Because of the position of the solid line below the high-temperature data in figure 1, the curve appears to be an incorrect fit. This is however an artifact of experiments performed to measure thermal conductivity at large temperature differences of materials whose conductivity is a nonlinear function of temperature. This situation is normal in measurements with the HPT method, which uses very large temperature differences. Use of the "thermal-conductivity integral" method reflects this state of affairs and correctly illustrates the relations between the individual data points and the thermal conductivity function. Here the functional

curve is concave upward.   As a consequence. any given data point,
which is closely related to the integral of the conductivity
function over the interval between the boundary temperatures, is
displaced upward [11, 12].

     The deviations of the individual data points from the curve
representing eq(9) are plotted in figure 2 and listed in the last
two columns of table 2 as absolute deviations (Dev) and as rela-
tive, or percentage, deviations (Kdev). Measurements on condition-
ed specimens by laboratories 2 and 4 (triangles) are indicated in
figures 1 and 2 by the filled triangles, and repeated measurements
by laboratory 6 (plus signs) are given by plus signs within
circles.   As discussed in references 11 and 12, experimental data
(Kdat) are actually averages of the thermal conductivity over the
interval from T_{low} (or T_1) to T_{high} (T_2).   Numbers in the Kcalc
column of table 2 were obtained by the thermal-conductivity
integral method, and represent likewise the temperature-averaged
mean of the conductivity function, eq(9), over the interval from
T_{low} to T_{high}.

     For the complete set of data the greatest positive deviation
from the fitted curve is 8.3 percent, and the greatest negative
deviation is -9.8 percent.   The standard deviation of the measure-
ments from the curve is 5.0 percent.

     One participant questioned whether this method of fitting is
fair, as three laboratories each submitted only four data points,
while one submitted eight data points.   In fact, the contribution
from each participant is weighted by the number of data points
taken.   This has the consequence that the sets of data from labora-
tories 1, 3, and 5 are each given only 50% of the weight of the set
from laboratory 6.   All other laboratories (submitting four, five,
or six data points) are also given only a fraction of the weight of
the one providing eight points.   In the matter of weighting data
points equally as opposed to weighting laboratories equally, I
agree that the point should be explicitly addressed, rather than
defaulting to an unexamined assumption. Both ways were tried.

     A new fit was performed over the same set of data, with
weightings chosen to give each laboratory equal weight, based on
"one man, one vote," rather than the choice of "one data point, one
vote" that led to eq(9).   The results of this fit yielded the
correlation

$$k = 18.184 + 0.036504\ T + 0.18478*10\text{-}6\ T^3 \qquad (10)$$

with k and T defined as before.   This functional relation is
plotted in figure 3.   The deviations of the data points from this
curve are plotted in figure 4 and listed in the last two columns of
table 3.   For this curve for equally weighted laboratories the

14

greatest positive deviation of the defining set of data is +8.5 percent and the greatest negative deviation is -9.2 percent, compared to deviations of +8.3 and -9.8 percent from the curve given by eq(9) for equally weighted data. The standard deviation here is 5.1 percent, as opposed to 5.0 percent for the equally weighted data. Thus for both methods of weighting data, the results are closely similar: greatest deviations of about +8.5 and -9.5 percent, and a standard deviation of 5 percent.

To estimate the precision of measurements performed by the HPT method, let us examine the set of data from only those participants that measured their specimens after conditioning (heating the specimens first to the highest temperature of measurement and then performing measurements. These data are from participants identified by code numbers 1, 2, 4 and 6 and are listed in table 4; the data are plotted in figure 5, encoded by symbols distinguishing the laboratories responsible for the data. The data from laboratory 2 are repeated measurements; data from laboratory 6 include both original data, which according to the submitted data sheet are valid, as well as additional data submitted in response to the call for repeated measurements. Laboratories 3,5 and 7, which had not submitted data for conditioned specimens, were apparently not able to perform repeated measurments. Thus there were four laboratories providing data on conditioned specimens.

By means of a least-squares fit a correlation of the same form as that of eq(9) was obtained. This correlation for the data from conditioned specimens is given by

$$k = 10.449 + 0.066314 \ T + 0.140223*10^{-6} \ T^3, \quad (11)$$

with k and T defined as before. This correlation is plotted as the solid line in figure 5. The data used to obtain this correlation cover the range in temperature from 318 to 652 K. Deviations of the individual data points from eq(10) are plotted in figure 6 and are listed in the last two columns of table 4.

The greatest positive and negative deviations of data points from the correlation (11) are both 6.4 percent; the total set of data fits the curve within a standard deviation of 4.3 percent. This standard deviation is considerably smaller than the standard deviation of the results from the interlaboratory comparison for the high-temperature GHPs [8], which covered a slightly greater temperature range (297 to 773 K), including the range used in this study. This comparison yielded standard deviations of 7.4 and 8 percent for the two materials studied, and maximum deviations of almost ±20 percent. Although we must not push the comparison between the GHP and the HPT methods too far, as there were only

15

seven participants in each of the two interlaboratory comparisons, comparisons between the precisions of the two methods might be made cautiously.

Having fitted only the data for conditioned specimens, obtaining eq(11), we now fit the set of data for nonconditioned specimens and compare the two sets. Table 5 lists the data for these nonconditioned specimens, which are plotted in figure 7. A weighted least-squares fit to the data for nonconditioned specimens gave the correlation

$$k = 42.980 - 0.05409 \; T + 0.34209*10^{-6} \; T^3, \qquad (12)$$

with k and T defined as before. This correlation is plotted as the solid line in figure 7.

The last two columns of table 5 show the deviations from the least-squares fit to these data, and the relative deviations are plotted in figure 8. The point of interest is that these data for nonconditioned specimens exhibit extremal deviations from the fitted curve of +6.9 and -7.5 percent. This is somewhat larger that the corresponding deviations of data for conditioned specimens from their fitted curve, as would be expected if the effect of conditioning is real. The standard deviation from the fitted curve for nonconditioned specimens is 4.7 percent, as opposed to 4.3 percent for the conditioned specimens.

The internal self-consistency of data for conditioned specimens seems to be better than that for nonconditioned specimens. We might conclude that conditioning makes a difference, but with reservations--there are only four sets of data for conditioned specimens and only five sets for nonconditioned specimens. The sizes of the two statistical samples are too small for us to be sure that the effect of conditioning is real.

The standard deviation of the complete set of thermal conductivity data, including measurements on both conditioned and nonconditioned specimens, is about 5 percent. The precision of the HPT method was estimated to be about 4.3 percent, so the effect of not conditioning the specimens seems to be about 1 percent.

Another instructive exercise is to compare the data for the nonconditioned specimens with the fitted curve for conditioned specimens; this curve is given analytically by eq (11). This was done by preserving the shape of the curve for conditioned specimens and simply raising it up or down parallel to the conductivity axis.

16

That is, we found a constant multiplier equal to 0.98, such that the rms deviations from this curve were minimized for data for nonconditioned specimens. The least-squares fit for this procedure is shown by the solid curve in figure 9 and the deviations are plotted in figure 10. The numerical values for this fit are given in table 6. The extremal deviations of the data for nonconditioned specimens were ± 9.7 percent, and the standard deviation of the fit is 5.4 percent.

This situation for a subset of the data appears paradoxical at first, with a standard deviation of 5.4 percent for the subset of nonconditioned specimens, compared with a standard deviation of only 4.95 for the set of all data, from the curve described by eq(9). This standard deviation of 5.4 percent may be understood to measure the internal consistency of data for purely nonconditioned specimens. The internal consistency of data for conditioned specimens, the complementary subset of data, is measured by a standard deviation of only 4.3 percent, which is less than the standard deviation of 4.95 for the complete set. The standard deviation for conditioned specimens is about 1 percent greater than that for nonconditioned specimens. Remembering that we deal here with a small statistical sample, we may again cautiously conclude that conditioning may make a difference of about 1 percent.

8. ESTIMATED PRECISION OF THE HPT METHOD

In measurement theory imprecision in a series of measurements of a physical quantity indicates the closeness of approach of the individual values to the mean value. Precision says nothing about how the mean value compares to the "true" value. Scatter of the measurements around the mean value is caused by random influences, which by definition have unpredictable magnitude and sign, and will tend to cancel out each other if a large enough number of measurements is made. The mean value of a large enough group of measurements of the same physical quantity tends to become free of the effects of the random influences.

By contrast, accuracy of a physical quantity indicates the closeness of approach of the mean of a series of measurements to the "true" value. Inaccuracy is caused by nonrandom, or systematic errors of measurement, which have not been corrected during the process of measurement because they are not known to the person performing the processes of measurement and analysis. These systematic errors, if present, might be disclosed by performing measurements of the same physical quantity on the same material but in a different type of apparatus, preferably based on a different principle of measurement. One could postulate that the true value is reached in the limit of a very large number of measurements, performed under very different conditions. If a large enough population of measurements has been obtained from many different types of apparatus that measure the same quantity, such that errors made in measurements based on one type of apparatus are offset by errors

17

in measurements from very different apparatuses, then the mean value of this large set of measurements may approach the "true" value. Accuracy of a set of measurements performed on one apparatus is then indicated by the difference between the mean value for the apparatus and the global mean for all apparatuses, that estimates the true value.

The present interlaboratory comparison of the HPT method involves instruments of the same type, so there may be inherent in this method some biases that have not been revealed by these measurements. We do not know of any comparative measurements of the same pipe insulation material on an apparatus of a different type. For the HPT method there is neither a theoretical value nor a standard reference material against which to compare the experimental results. Therefore the "true" value of the material measured here is unknown. An estimate of accuracy of the horizontal pipe-test method cannot be made at this time.

The precision of measurements with the HPT method is indicated by the scatter of measurement data around the group mean. One estimate of the group mean of the thermal conductivity is given by the fitted curve, eq (11), for conditioned specimens measured by four different laboratories. An estimate of the scatter of the data around the mean for the four laboratories is given by the standard deviation of the data (4.3 percent) from the fitted curve. A larger statistical sample for basing an estimate of accuracy is available: the complete set of data including both conditioned and nonconditioned specimens. For this set the standard deviation, or scatter of data about the mean, is about 5 percent. Preferring to be conservative by basing the estimate on the largest possible set of data and by choosing the looser of the two possible estimates, I estimate the precision of the HPT method to be 5 percent over the range of temperature from 318 to 530 K. This is based on measurements performed with only one type of apparatus, the guarded-end HPT apparatus, and only one type of insulation material. Caution should be exercised in generalizing these results to another type of HPT apparatus.

Because the participants in this interlaboratory comparison did not measure the same specimen, but rather measured different specimens (obtained from the same supplier, to reduce variability), some of the scatter of the measurements about the mean, or imprecision, may be due to material variability in the heat-transfer properties of the different specimens used.

9. SUMMARY AND CONCLUSIONS

Measurements of thermal conductivity have been presented for a mineral-fiber pipe insulation material at mean temperatures from 318 to 652 K. Two methods of fitting the data were used: weighting individual data points equally, and weighting individual laboratories equally. There seems to be no significant difference in the closeness of the fits for the two different methods of weighting.

Of seven participating laboratories, four responded with data on specimens that were conditioned by heat first to the highest temperature of measurement, so that physical changes in the specimen had already occurred before data was taken. This conditioning was an issue that led to submission of additional measurements by the participants. Results from these laboratories, compared with measurements from those that did not do so, have suggested that conditioning the specimens before performing measurements may have a detectable effect on the measured thermal conductivity. The small number of participants makes it statistically difficult to draw firm conclusions on this point.

Reference equations are obtained which describe the dependence of the thermal conductivity on temperature for the refractory pipe insulation material, and are illustrated in the figures. Figures 1 and 2 compare results from all seven participants. Figures 3 and 4 make a similar comparison, with equal weighting for each participant. Figures 5 and 6 give the thermal conductivity of conditioned specimens. Figures 7 through 10 allow comparisons to be made with specimens that were not so conditioned. All the odd-numbered figures illustrate the fact that data points from measurements of thermal conductivity with large temperature differences do not coincide with the thermal conductivity function at the same temperature. This shows the necessity of using the thermal conductivity integral method [11,12] to obtain the heat transport to be expected under large differences in temperature between boundaries.

The reference equations are used to analyze the precision of interlaboratory measurements made by the seven laboratories that participated in the round robin. The precision of measurement of thermal conductivity by the horizontal pipe-test method is estimated to be about 5 percent as measured by the standard deviation of measurements about the group mean for the participants. This estimate is almost independent of the effects of conditioning. An estimate of the accuracy of measurements performed with the HPT apparatus cannot be made at this time.

The comparison of the guarded-hot-plate method with the pipe-test method in Section 2 of this report suggest qualitatively that there may be fewer uncorrected biases in measurements made with the HPT apparatus than with the GHP at high temperatures. In contrast to the comparative method, which intrinsically depends on one or two reference materials in order to determine the conductivity of the specimen under study, both the HPT and the GHP involve "absolute" methods of measurement that are in principle independent of calibration standards. This interlaboratory comparison of the HPT method demonstrated that for a population of seven laboratories using both conditioned and nonconditioned specimens, the standard deviation of the data from the group mean was about 4.5 percent over a range of mean specimen temperatures up to 652 K. Using only conditioned specimens over the same range of mean temperatures, four participants achieved a standard deviation from the group mean of only 4.3 percent. This is significantly better than the standard deviation of almost 8 percent resulting for the interlaboratory comparison of the GHP method at high temperatures. Although there is at present no standard reference material for cross-checking measurements made with the HPT apparatus, current evidence suggest that results of measurements with the HPT are on the average at least as good as those made with the GHP, both apparatuses being in principle "absolute" methods. With both instruments, care in experimental design and technique is indispensable in obtaining trustworthy data.

A very desirable next step would be to perform an interlaboratory test comparing the horizontal pipe-test apparatus and the guarded hot plate, on the same specimens. Because of the great difference in geometry inherent in these two measurement methods, it would be almost impossible to use identical specimens on the two different types of apparatus. However the use of loose-fill insulation materials, properly conditioned by standardized procedures, could permit the comparison of results from these two different types of apparatus. To compare measurements from these apparatuses is highly desirable, since the GHP apparatus when used at temperatures significantly higher than ambient seems to fall short of expectations. The limited information from two interlaboratory comparisons, each involving only seven laboratories, suggests that the HPT apparatus is more reliable at high temperatures than is the GHP. Yet the HPT method is limited to materials with cylindrical geometry, and cannot be used to measure a large fraction of the types of commercial insulation materials, in flat or block geometry, of great economic importance in residential building construction.

10. REFERENCES

[1]. Siu, M.C.I. and Bulik, C. "National Bureau of Standards Line-Heat-Source Guarded-Hot-Plate Apparatus", Rev. Sci. Instrum. 52/11, 1709-1716 (1981).

[2]. Smith, David R., Hust, J.G. and Van Poolen, Lambert J. "A Guarded-Hot-Plate Apparatus for Measuring Effective Thermal Conductivity of Insulations Between 80 K and 360 K", NBSIR 81-1657 (U.S. National Bureau of Standards, January 1982).

[3]. Rennex, Brian. "Error Analysis for the National Bureau of Standards 1016 mm Guarded Hot Plate", NBSIR 83-2674 (U.S. National Bureau of Standards, February 1983).

[4]. Hust, J.G., Filla, B.J., Hurley, J.A. and Smith, D.R. "An Automated High-Temperature Guarded-Hot-Plate Apparatus for Measuring Apparent Thermal Conductivity", NBSIR 88-3089 (U.S. National Bureau of Standards, May 1988).

[5]. Hust, J. G. and Pelanne, C. M. "Round Robins on the Apparent Thermal Conductivity of Low-Density Glass Fiber Insulations Using Guarded Hot Plate and Heat Flow Meter Apparatus", NBSIR 85-3026 (U.S. National Bureau of Standards, 1985)

[6]. ASTM Standard Test Method C 177 for "STEADY-STATE HEAT FLUX MEASUREMENTS AND THERMAL TRANSMISSION PROPERTIES BY MEANS OF THE GUARDED-HOT-PLATE APPARATUS", ANNUAL BOOK OF ASTM STANDARDS, Vol. 04.06, pp. 21-36 (ASTM, Philadelphia, 1986).

[7]. ASTM Standard Test Method C 335 for "STEADY-STATE HEAT TRANSFER PROPERTIES OF HORIZONTAL PIPE INSULATION", ANNUAL BOOK OF ASTM STANDARDS, Vol. 04.06, pp. 83-96 (ASTM, Philadelphia, 1986).

[8]. Kimball, L.R. "Thermal Conductance of Pipe Insulation--A Large-Scale Test Apparatus", Heat Transmission Measurements in Thermal Insulations, ASTM STP 544, pp. 135-146, (ASTM, Philadelphia, 1974).

[9]. Hollingsworth, M. Jr. "An Interlaboratory Comparison of the ASTM C 335 Pipe Insulation Test", Thermal Transmission Measurements of Insulation, ASTM STP 660, R.P. Tye, Ed., pp. 50-59, (ASTM, Philadelphia, 1978).

[10]. Hust, Jerome G. and Smith, David R. "Round-Robin Measurements of the Apparent Thermal Conductivity of Two Refractory Insulation Materials, Using High-Temperature Guarded-Hot-Plate Apparatus", NBSIR 88-3087 (U.S. National Bureau of Standards, April 1988).

[11]. Hust, J. G. and Lankford, A. B. "Comments on the Measurement of Thermal Conductivity and Presentation of a Thermal Conductivity Integral Method", International Journal of Thermophysics, 3/1, 67-77, (1982).

[12]. ASTM Standard Test Method C 1045 for "CALCULATING THERMAL TRANSMISSION PROPERTIES FROM STEADY-STATE HEAT FLUX MEASUREMENTS", ANNUAL BOOK OF ASTM STANDARDS, Vol. 04.06, pp. 689-696 (ASTM, Philadelphia, 1986).

Table 2. Interlaboratory comparison of apparent thermal conductivity of high-temperature mineral-fiber pipe insulation measured on seven different horizontal pipe-test apparatuses. The nominal density of the specimens is 90 kg/m³ (5.6 lb /ft³. Specimens had a nominal inner diameter of 89 mm (3.5 in), and a wall thickness of 51 mm (2 in). Measurements were performed at the ambient atmospheric pressure of each laboratory, 83 to 100 kPa (630 to 760 Torr), and at mean temperatures from 318 to 652 K (113 to 1238°F). Kcalc is computed from eq (9). The standard deviation of the data set from eq (9) for all specimens is 4.95 percent.

Lab No.	Density (kg/m³)	Thigh (C)	Tlow (C)	DelT (C)	Tave (C)	Kcalc mW/(m.K)	Kdat mW/(m.K)	Dev	Kdev (Pct)
1	100.91	942.928	360.983	581.944	651.956	103.649	100.294	-3.36	-3.24
1	100.91	854.483	349.039	505.444	601.761	87.912	84.010	-3.90	-4.44
1	100.91	687.094	328.261	358.833	507.678	64.382	62.828	-1.55	-2.41
1	100.91	503.706	309.650	194.056	406.678	46.497	45.536	-0.96	-2.07
2	86.98	379.261	302.150	77.111	340.706	38.167	38.907	0.74	1.94
2	86.98	419.206	306.150	113.056	362.678	40.667	40.924	0.26	0.63
2	86.98	523.706	313.817	209.889	418.761	48.251	49.426	1.18	2.44
2	86.98	632.928	323.928	309.000	478.428	58.444	61.531	3.09	5.28
2	85.60	624.706	321.706	303.000	473.206	57.489	61.819	4.33	7.53
2	85.60	526.094	310.983	215.111	418.539	48.262	51.156	2.89	6.00
2	85.60	422.706	304.817	117.889	363.761	40.808	42.510	1.70	4.17
2	85.60	379.372	302.428	76.944	340.900	38.187	39.483	1.30	3.39
3	94.35	339.261	299.039	40.222	319.150	35.934	38.907	2.97	8.27
3	94.35	451.483	306.483	145.000	378.983	42.706	44.671	1.96	4.60
3	94.35	590.928	318.706	272.222	454.817	54.150	56.199	2.05	3.78
3	94.35	728.150	334.817	393.333	531.483	69.596	73.491	3.90	5.60
4	92.26	439.706	303.761	135.944	371.733	41.804	40.492	-1.31	-3.14
4	92.26	529.761	311.039	218.722	420.400	48.550	47.553	-1.00	-2.05
4	92.26	628.483	320.650	307.833	474.567	57.779	57.496	-0.28	-0.49
4	92.26	726.539	332.928	393.611	529.733	69.250	68.303	-0.95	-1.37
4	92.26	493.039	308.428	184.611	400.733	45.652	44.527	-1.12	-2.46
5	90.13	404.283	297.822	106.461	351.053	39.354	36.601	-2.75	-7.00
5	90.13	429.311	302.256	127.056	365.783	41.076	37.466	-3.61	-8.79
5	90.13	502.089	306.483	195.606	404.286	46.192	41.645	-4.55	-9.84
5	90.13	551.383	312.694	238.689	432.039	50.377	45.680	-4.70	-9.32
6	90.50	549.561	310.639	238.922	430.100	50.099	50.147	0.05	0.10
6	90.50	438.889	303.800	135.089	371.344	41.754	39.772	-1.98	-4.75
6	90.50	348.761	298.617	50.144	323.689	36.390	35.305	-1.09	-2.98
6	88.23	727.850	329.840	398.010	528.845	69.156	73.549	4.39	6.35
6	88.23	533.100	309.600	223.500	421.350	48.722	52.193	3.47	7.12
6	88.23	338.580	296.950	41.630	317.765	35.802	38.345	2.54	7.10
7	90.50	375.650	300.261	75.389	337.956	37.877	38.042	0.17	0.44
7	90.50	438.761	303.872	134.889	371.317	41.750	41.933	0.18	0.44
7	90.50	493.539	308.428	185.111	400.983	45.688	46.256	0.57	1.24
7	90.50	559.761	314.428	245.333	437.094	51.179	52.308	1.13	2.21
7	90.50	679.150	327.150	352.000	503.150	63.434	65.421	1.99	3.13

Standard Deviation = 4.95

Table 3.   Interlaboratory comparison of apparent thermal conductivity of high-temperature mineral-fiber pipe insulation measured on seven different horizontal pipe-test apparatuses. Kcalc is computed from eq (10), obtained by weighting each laboratory equally.   The standard deviation of the data set from eq (10) for all specimens is 5.08 percent.

Lab No.	Density (kg/m^3)	Thigh (C)	Tlow (C)	DelT (C)	Tave (C)	Kcalc mW/(m.K)	Kdat mW/(m.K)	Dev	Kdev (Pct)
1	100.91	942.928	360.983	581.944	651.956	103.387	100.294	-3.09	-2.99
1	100.91	854.483	349.039	505.444	601.761	87.518	84.010	-3.51	-4.01
1	100.91	687.094	328.261	358.833	507.678	63.914	62.828	-1.09	-1.70
1	100.91	503.706	309.650	194.056	406.678	46.165	45.536	-0.63	-1.36
2	86.98	379.261	302.150	77.111	340.706	38.023	38.907	0.88	2.33
2	86.98	419.206	306.150	113.056	362.678	40.452	40.924	0.47	1.17
2	86.98	523.706	313.817	209.889	418.761	47.892	49.426	1.53	3.20
2	86.98	632.928	323.928	309.000	478.428	57.994	61.531	3.54	6.10
2	85.60	624.706	321.706	303.000	473.206	57.045	61.819	4.77	8.37
2	85.60	526.094	310.983	215.111	418.539	47.905	51.156	3.25	6.79
2	85.60	422.706	304.817	117.889	363.761	40.590	42.510	1.92	4.73
2	85.60	379.372	302.428	76.944	340.900	38.042	39.483	1.44	3.79
3	94.35	339.261	299.039	40.222	319.150	35.865	38.907	3.04	8.48
3	94.35	451.483	306.483	145.000	378.983	42.445	44.671	2.23	5.25
3	94.35	590.928	318.706	272.222	454.817	53.728	56.199	2.47	4.60
3	94.35	728.150	334.817	393.333	531.483	69.125	73.491	4.37	6.32
4	92.26	439.706	303.761	135.944	371.733	41.563	40.492	-1.07	-2.58
4	92.26	529.761	311.039	218.722	420.400	48.189	47.553	-0.64	-1.32
4	92.26	628.483	320.650	307.833	474.567	57.334	57.496	0.16	0.28
4	92.26	726.539	332.928	393.611	529.733	68.781	68.303	-0.48	-0.69
4	92.26	493.039	308.428	184.611	400.733	45.334	44.527	-0.81	-1.78
5	90.13	404.283	297.822	106.461	351.053	39.177	36.601	-2.58	-6.57
5	90.13	429.311	302.256	127.056	365.783	40.853	37.466	-3.39	-8.29
5	90.13	502.089	306.483	195.606	404.286	45.867	41.645	-4.22	-9.20
5	90.13	551.383	312.694	238.689	432.039	49.994	45.680	-4.31	-8.63
6	90.50	549.561	310.639	238.922	430.100	49.720	50.147	0.43	0.86
6	90.50	438.889	303.800	135.089	371.344	41.515	39.772	-1.74	-4.20
6	90.50	348.761	298.617	50.144	323.689	36.304	35.305	-1.00	-2.75
6	88.22	727.850	329.840	398.010	528.845	68.689	73.560	4.87	7.09
6	88.22	533.100	309.600	223.500	421.350	48.360	52.210	3.85	7.96
6	88.22	338.580	296.950	41.630	317.765	35.738	38.360	2.62	7.34
7	90.50	375.650	300.261	75.389	337.956	37.742	38.042	0.30	0.80
7	90.50	438.761	303.872	134.889	371.317	41.511	41.933	0.42	1.02
7	90.50	493.539	308.428	185.111	400.983	45.370	46.256	0.89	1.95
7	90.50	559.761	314.428	245.333	437.094	50.786	52.308	1.52	3.00
7	90.50	679.150	327.150	352.000	503.150	62.968	65.421	2.45	3.90

Standard Deviation =   5.08

Table 4. Interlaboratory comparison of apparent thermal conductivity of high-temperature mineral-fiber pipe insulation. This set of specimens is restricted to only those which were conditioned by heating them to the highest temperature of measurement before data was taken. Kcalc is computed from eq (11). The standard deviation of the data set from eq (11) for conditioned specimens is 4.34 percent.

Lab No.	Density (kg/m^3)	Thigh (C)	Tlow (C)	DelT (C)	Tave (C)	Kcalc mW/(m.K)	Kdat mW/(m.K)	Dev	Kdev (Pct)
1	100.91	942.928	360.983	581.944	651.956	100.280	100.294	0.01	0.01
1	100.91	854.483	349.039	505.444	601.761	86.299	84.010	-2.29	-2.65
1	100.91	687.094	328.261	358.833	507.678	64.754	62.828	-1.93	-2.98
1	100.91	503.706	309.650	194.056	406.678	47.386	45.536	-1.85	-3.90
2	85.60	624.706	321.706	303.000	473.206	58.210	61.819	3.61	6.20
2	85.60	526.094	310.983	215.111	418.539	49.164	51.156	1.99	4.05
2	85.60	422.706	304.817	117.889	363.761	41.498	42.510	1.01	2.44
2	85.60	379.372	302.428	76.944	340.900	38.681	39.483	0.80	2.07
4	92.26	726.539	332.928	393.611	529.733	69.299	68.303	-1.00	-1.44
4	92.26	493.039	308.428	184.611	400.733	46.526	44.527	-2.00	-4.30
6	90.50	549.561	310.639	238.922	430.100	50.988	50.147	-0.84	-1.65
6	90.50	438.889	303.800	135.089	371.344	42.492	39.772	-2.72	-6.40
6	90.50	348.761	298.617	50.144	323.689	36.698	35.305	-1.39	-3.80
6	88.23	727.850	329.840	398.010	528.845	69.195	73.549	4.35	6.29
6	88.23	533.100	309.600	223.500	421.350	49.618	52.193	2.58	5.19
6	88.23	338.580	296.950	41.630	317.765	36.040	38.345	2.31	6.40

Standard Deviation = 4.34

Table 5.   Interlaboratory comparison of apparent thermal conductivity of high-temperature mineral-fiber pipe insulation. This set of specimens is restricted to only those which were not conditioned by heating them to the highest temperatures of measurement before data was taken.   Kcalc is computed from eq (12).   The standard deviation of the data set from eq (12) for nonconditioned specimens is 4.72 percent.

Lab No.	Density (kg/m³)	Thigh (C)	Tlow (C)	DelT (C)	Tave (C)	Kcalc mW/(m.K)	Kdat mW/(m.K)	Dev	Kdev (Pct)
2	86.98	379.261	302.150	77.111	340.706	38.252	38.907	0.65	1.71
2	86.98	419.206	306.150	113.056	362.678	40.077	40.924	0.85	2.12
2	86.98	523.706	313.817	209.889	418.761	47.026	49.426	2.40	5.10
2	86.98	632.928	323.928	309.000	478.428	58.468	61.531	3.06	5.24
3	94.35	339.261	299.039	40.222	319.150	36.880	38.907	2.03	5.50
3	94.35	451.483	306.483	145.000	378.983	41.781	44.671	2.89	6.92
3	94.35	590.928	318.706	272.222	454.817	53.444	56.199	2.76	5.16
3	94.35	728.150	334.817	393.333	531.483	72.620	73.491	0.87	1.20
4	92.26	439.706	303.761	135.944	371.733	41.031	40.492	-0.54	-1.31
4	92.26	529.761	311.039	218.722	420.400	47.376	47.553	0.18	0.37
4	92.26	628.483	320.650	307.833	474.567	57.716	57.496	-0.22	-0.38
4	92.26	726.539	332.928	393.611	529.733	72.195	68.303	-3.89	-5.39
5	90.13	404.283	297.822	106.461	351.053	39.130	36.601	-2.53	-6.46
5	90.13	429.311	302.256	127.056	365.783	40.440	37.466	-2.97	-7.35
5	90.13	502.089	306.483	195.606	404.286	45.038	41.645	-3.39	-7.53
5	90.13	551.383	312.694	238.689	432.039	49.301	45.680	-3.62	-7.35
7	90.50	375.650	300.261	75.389	337.956	38.067	38.042	-0.02	-0.06
7	90.50	438.761	303.872	134.889	371.317	40.985	41.933	0.95	2.31
7	90.50	493.539	308.428	185.111	400.983	44.519	46.256	1.74	3.90
7	90.50	559.761	314.428	245.333	437.094	50.152	52.308	2.16	4.30
7	90.50	679.150	327.150	352.000	503.150	64.668	65.421	0.75	1.16

Standard Deviation = 4.72

Table 6. Apparent thermal conductivity of specimens of high-temperature mineral-fiber pipe insulation that were not conditioned before they were measured. The last two columns indicate deviations of these data from the least-squares curve, eq (11), defined by specimens that were conditioned before they were measured. The standard deviation of the data set from eq (11) for conditioned specimens is 5.40 percent.

| Lab No.	Density (kg/m^3)	Thigh (C)	Tlow (C)	DelT (C)	Tave (C)	Kcalc mW/(m.K)	Kdat mW/(m.K)	Dev	Kdev (Pct)
2	86.98	379.261	302.150	77.111	340.706	37.881	38.907	1.03	2.71
2	86.98	419.206	306.150	113.056	362.678	40.519	40.924	0.41	1.00
2	86.98	523.706	313.817	209.889	418.761	48.173	49.426	1.25	2.60
2	86.98	632.928	323.928	309.000	478.428	57.942	61.531	3.59	6.19
3	94.35	339.261	299.039	40.222	319.150	35.461	38.907	3.45	9.72
3	94.35	451.483	306.483	145.000	378.983	42.617	44.671	2.05	4.82
3	94.35	590.928	318.706	272.222	454.817	53.877	56.199	2.32	4.31
3	94.35	728.150	334.817	393.333	531.483	68.226	73.491	5.26	7.72
4	92.26	439.706	303.761	135.944	371.733	41.687	40.492	−1.20	−2.87
4	92.26	529.761	311.039	218.722	420.400	48.455	47.553	−0.90	−1.86
4	92.26	628.483	320.650	307.833	474.567	57.305	57.496	0.19	0.33
4	92.26	726.539	332.928	393.611	529.733	67.904	68.303	0.40	0.59
5	90.13	404.283	297.822	106.461	351.053	39.131	36.601	−2.53	−6.46
5	90.13	429.311	302.256	127.056	365.783	40.934	37.466	−3.47	−8.47
5	90.13	502.089	306.483	195.606	404.286	46.119	41.645	−4.47	−9.70
5	90.13	551.383	312.694	238.689	432.039	50.238	45.680	−4.56	−9.07
7	90.50	375.650	300.261	75.389	337.956	37.568	38.042	0.47	1.26
7	90.50	438.761	303.872	134.889	371.317	41.633	41.933	0.30	0.72
7	90.50	493.539	308.428	185.111	400.983	45.625	46.256	0.63	1.38
7	90.50	559.761	314.428	245.333	437.094	51.018	52.308	1.29	2.53
7	90.50	679.150	327.150	352.000	503.150	62.576	65.421	2.85	4.55

Standard Deviation = 5.40

27
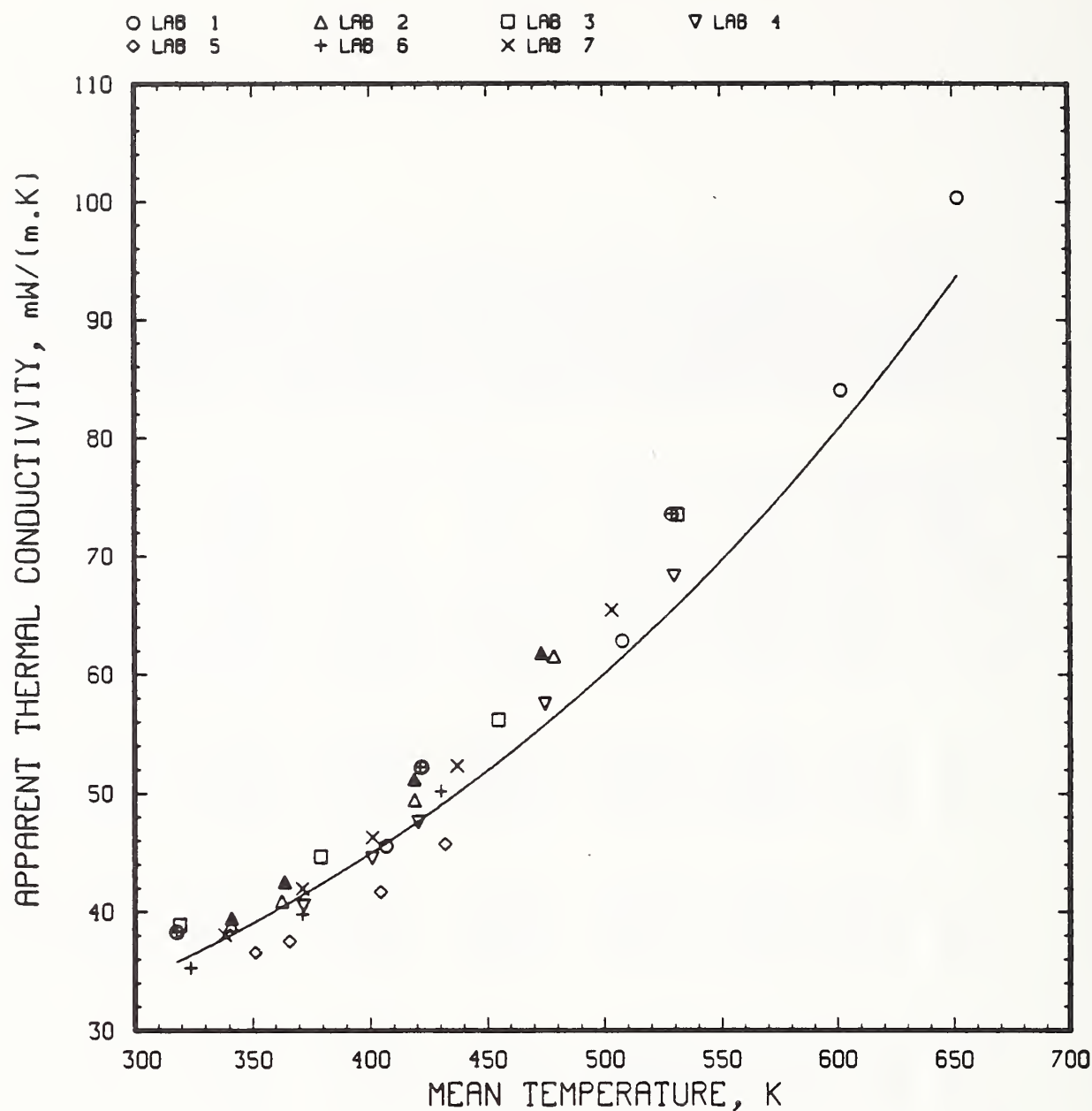
Figure 1. Interlaboratory comparison of apparent thermal conductivity of
————— mineral-fiber pipe insulation measured on the horizontal pipe-
test apparatus. The nominal density of the specimens is 90 kg/m³ (5.6
lb /ft³). Measurements were performed in air at ambient pressure. The
solid line is a plot of eq (9), thermal conductivity as a function of
temperature. Each point on this line represents the thermal conductivity
determined by the local thermal gradient (vanishing temperature differ-
ence), at a point within the specimen; the temperature at that point is
given by the value from the temperature axis. Each symbol represents the
experimental conductivity obtained at (large) non-zero temperature dif-
ferences between the boundary surfaces at Tcold and Thot. The value of
conductivity at the symbol is the ratio of the integral of the conducti-
vity function between Tcold and Thot to the corresponding temperature
difference. For this reason the data points deviate from the curve when
large temperature differences are used. Specimens from laboratories 1, 2,
4 and 6 were conditioned by heating specimens to the highest temperatures
of measurement before taking data. Filled triangles denote repeated meas-
urements by laboratory 2 and circled "plus" signs denote repeat measure-
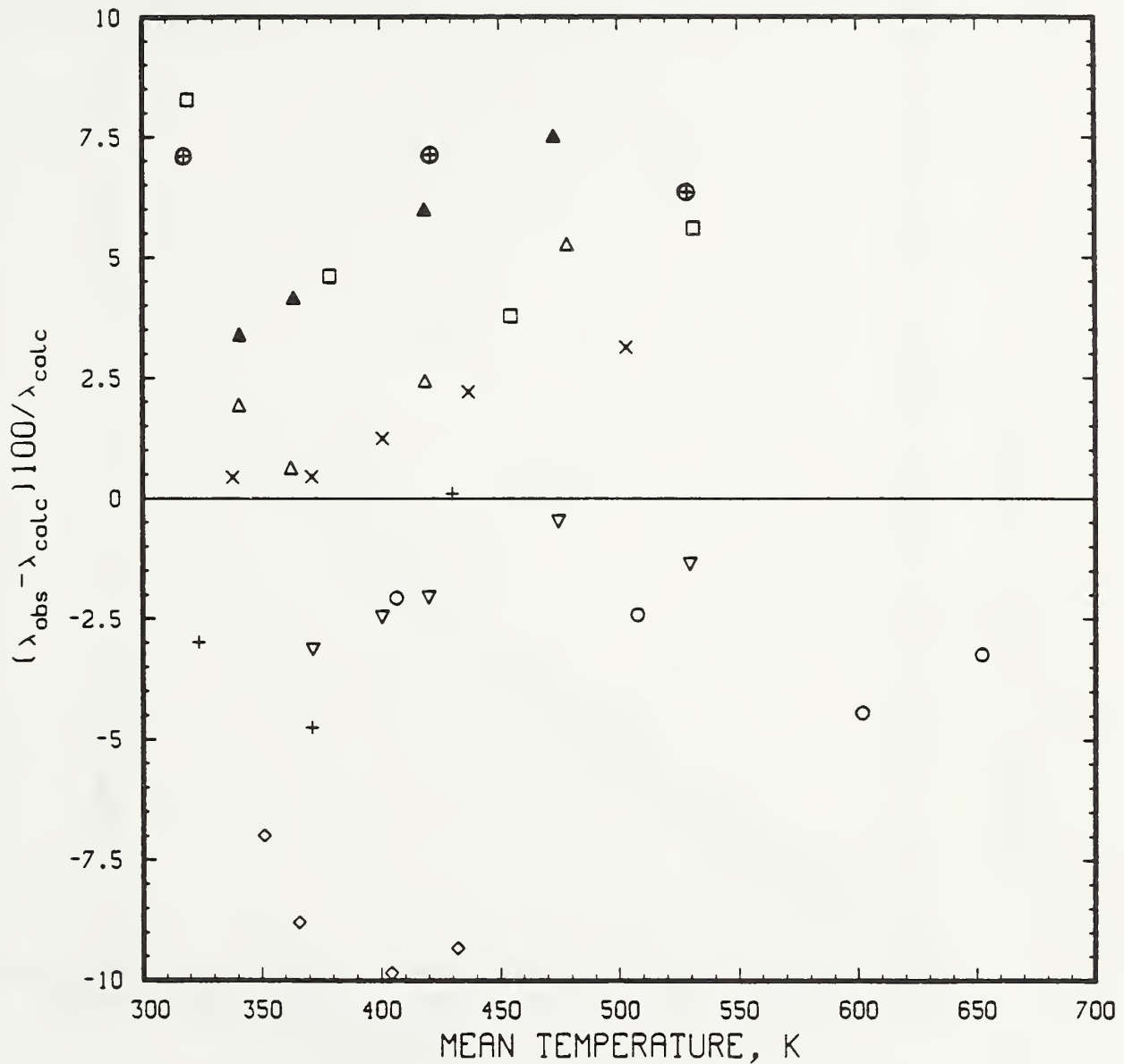ments by laboratory 6.

28

Figure 2. Deviations of apparent thermal conductivity of mineral-fiber
—————— pipe insulation, as measured by all seven participants, from
values calculated with eq (9), thermal conductivity as a function of
temperature. Each experimental conductivity value is obtained at (large)
nonzero temperature differences between the boundary surfaces at Tcold
and Thot. The integral of the conductivity function is computed over the
temperature interval from Tcold to Thot. We compute the ratio of this
integral to the corresponding temperature difference to obtain the mean
conductivity over the interval from Tcold to Thot. The difference between
the computed mean and the experimental value is the deviation. Filled
triangles denote repeated measurements by laboratory 2, and circled plus
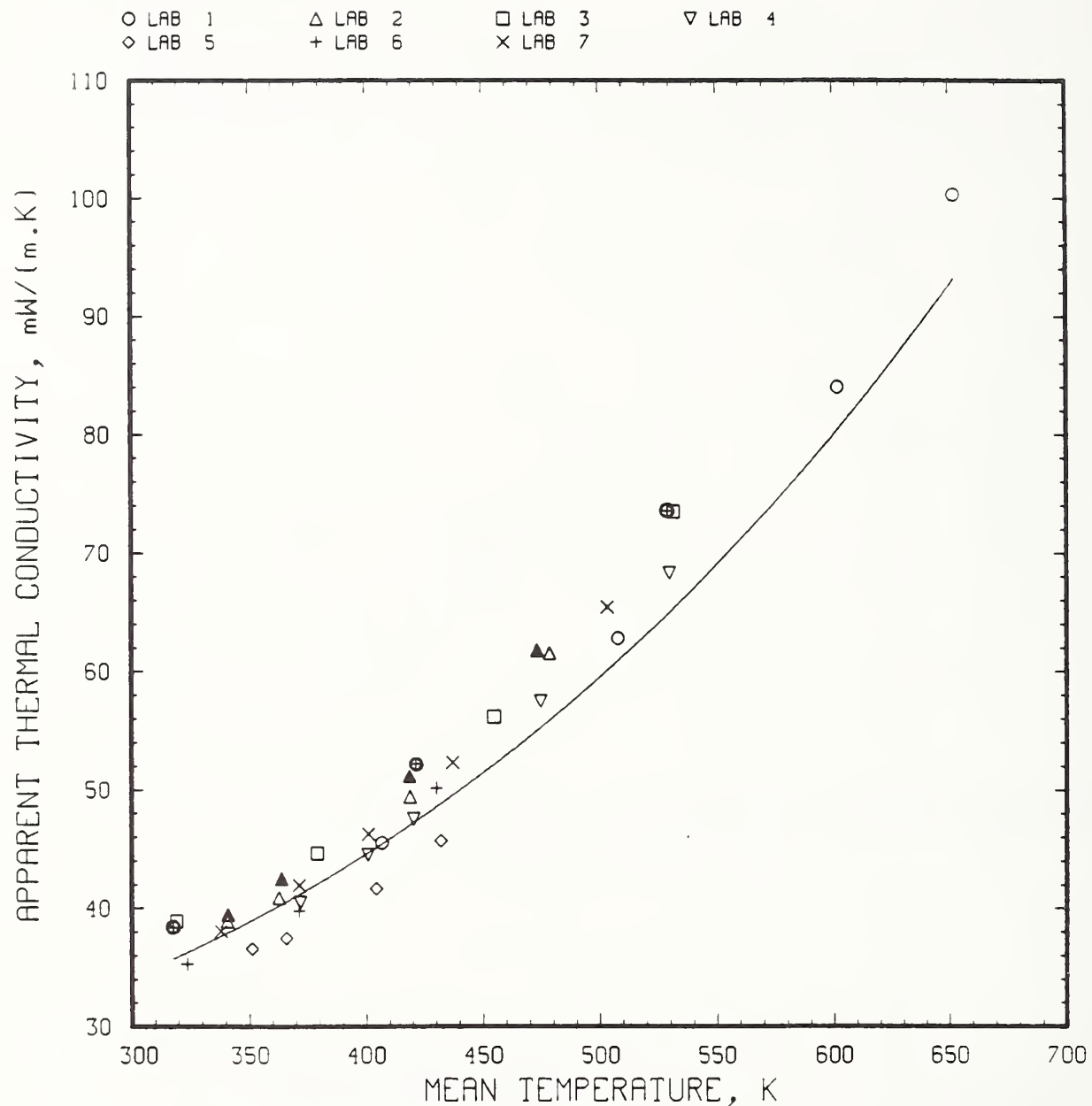signs denote repeated measurements by laboratory 6.

29

Figure 3. Interlaboratory comparison of apparent thermal conductivity of
————————— mineral—fiber pipe insulation measured on the horizontal pipe—
test apparatus. Measurements were performed in air at ambient pressure.
The solid line is a plot of eq (10), for thermal conductivity as a function
of temperature, obtained by weighting each laboratory equally. Specimens
from laboratories 1, 2, 4, and 6 were conditioned by heating specimens
to the highest temperatures of measurement before taking data. Filled tri—
angles denote repeated measurements by laboratory 2 and circled plus signs
denote repeated measurements by laboratory 6. Please see the caption of
figure 1 for an explanation of the difference between the experimental
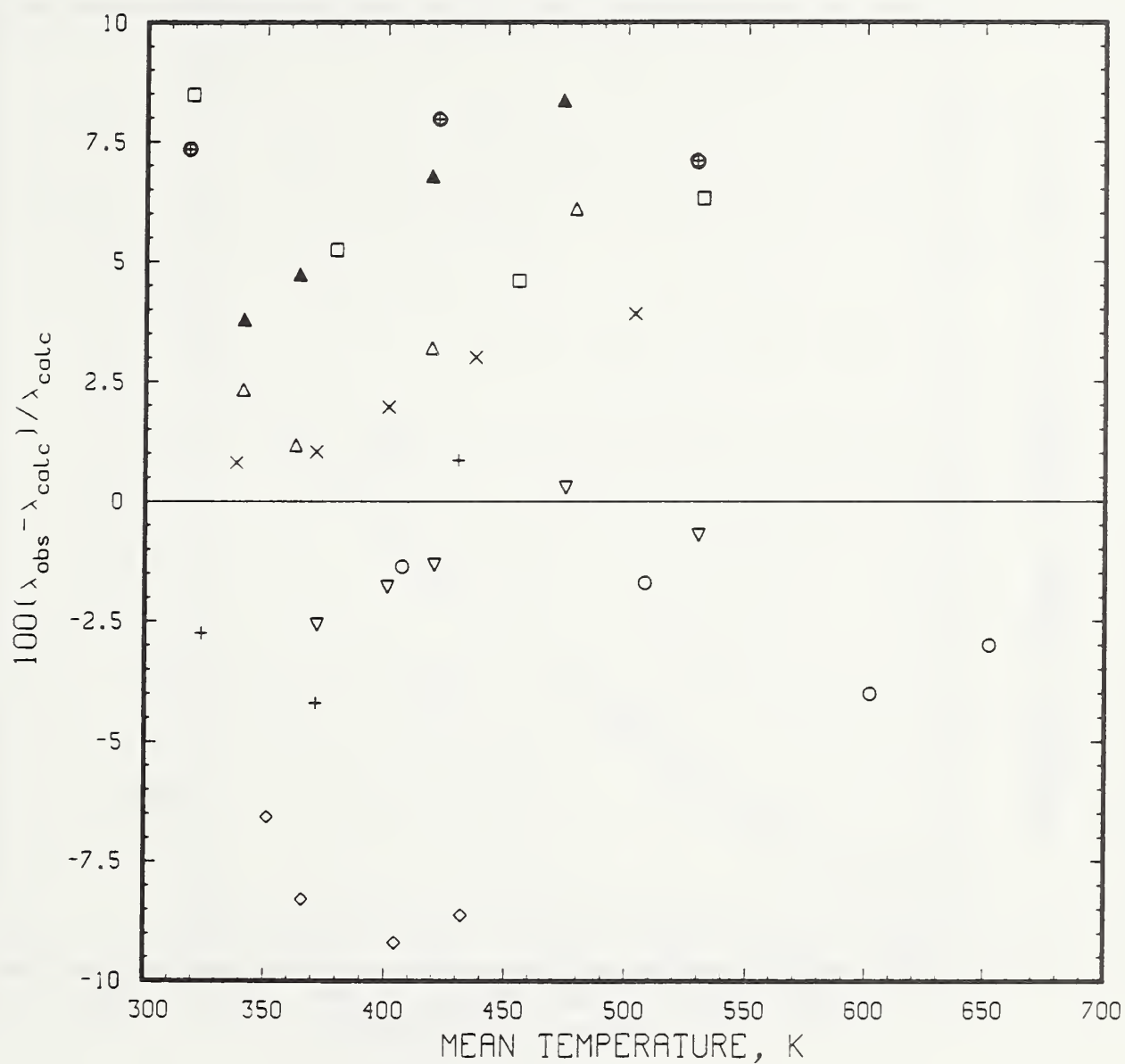points (symbols) and the fitted conductivity function.

Figure 4. Deviations of apparent thermal conductivity of mineral-fiber pipe insulation, as measured by all seven participants, from values calculated with eq (10) obtained by weighting each _laboratory_ equally. Filled triangles denote repeated measurements by laboratory 2, and circled plus signs denote repeat measurements by laboratory 6. Please see caption of figure 2 for an explanation of how the deviation is computed.
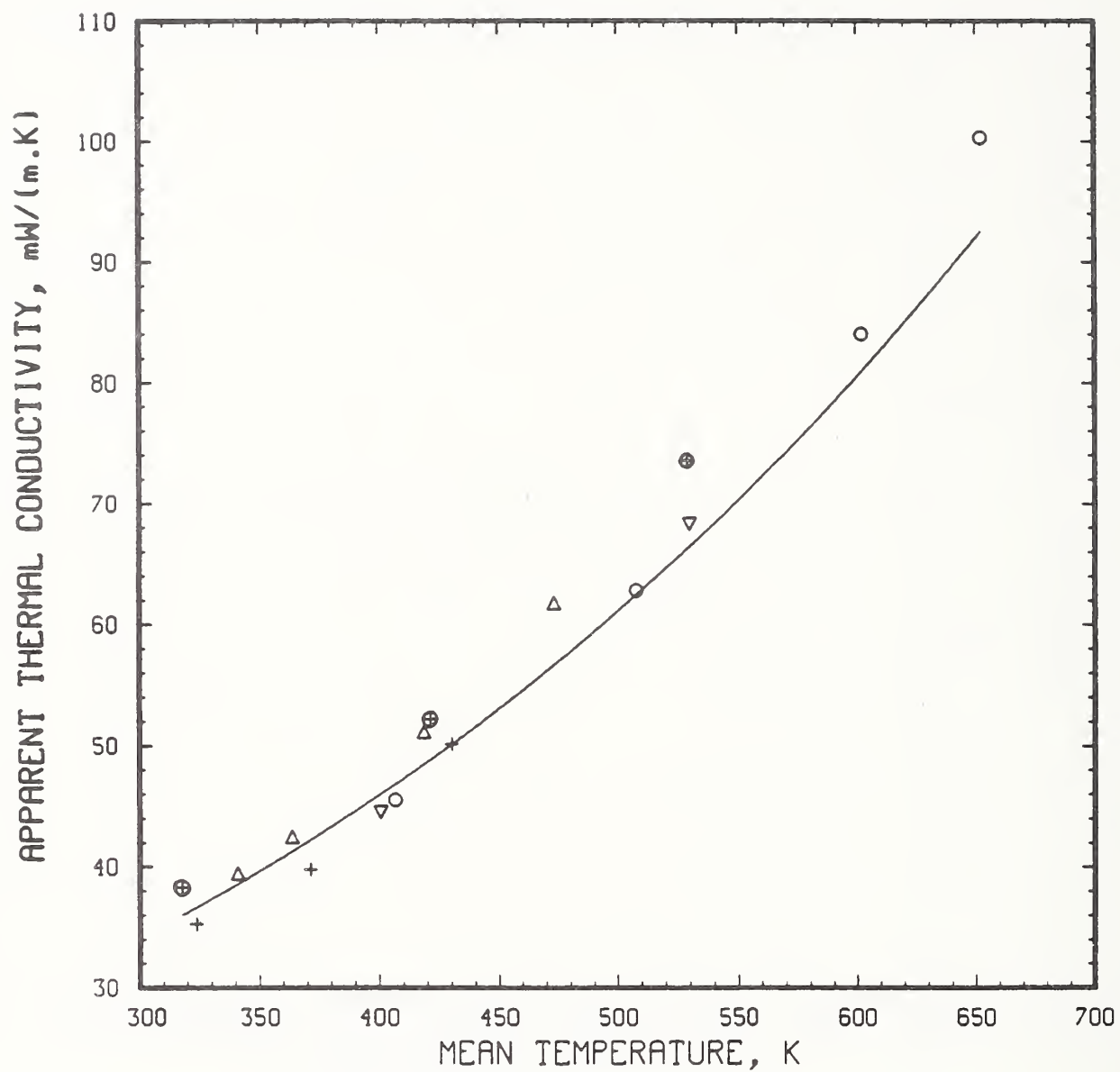
Figure 5. Interlaboratory comparison of apparent thermal conductivity
—————— of mineral-fiber pipe insulation measured on the horizontal pipe-
test apparatus. Measurements were performed in air at ambient pressure.
Specimens were conditioned by heating them to the highest temperatures of
measurement before taking data. The solid line is a plot of eq (11) for
thermal conductivity as a function of temperature. Please see the caption
of figure 1 for an explanation of the difference between the experimental
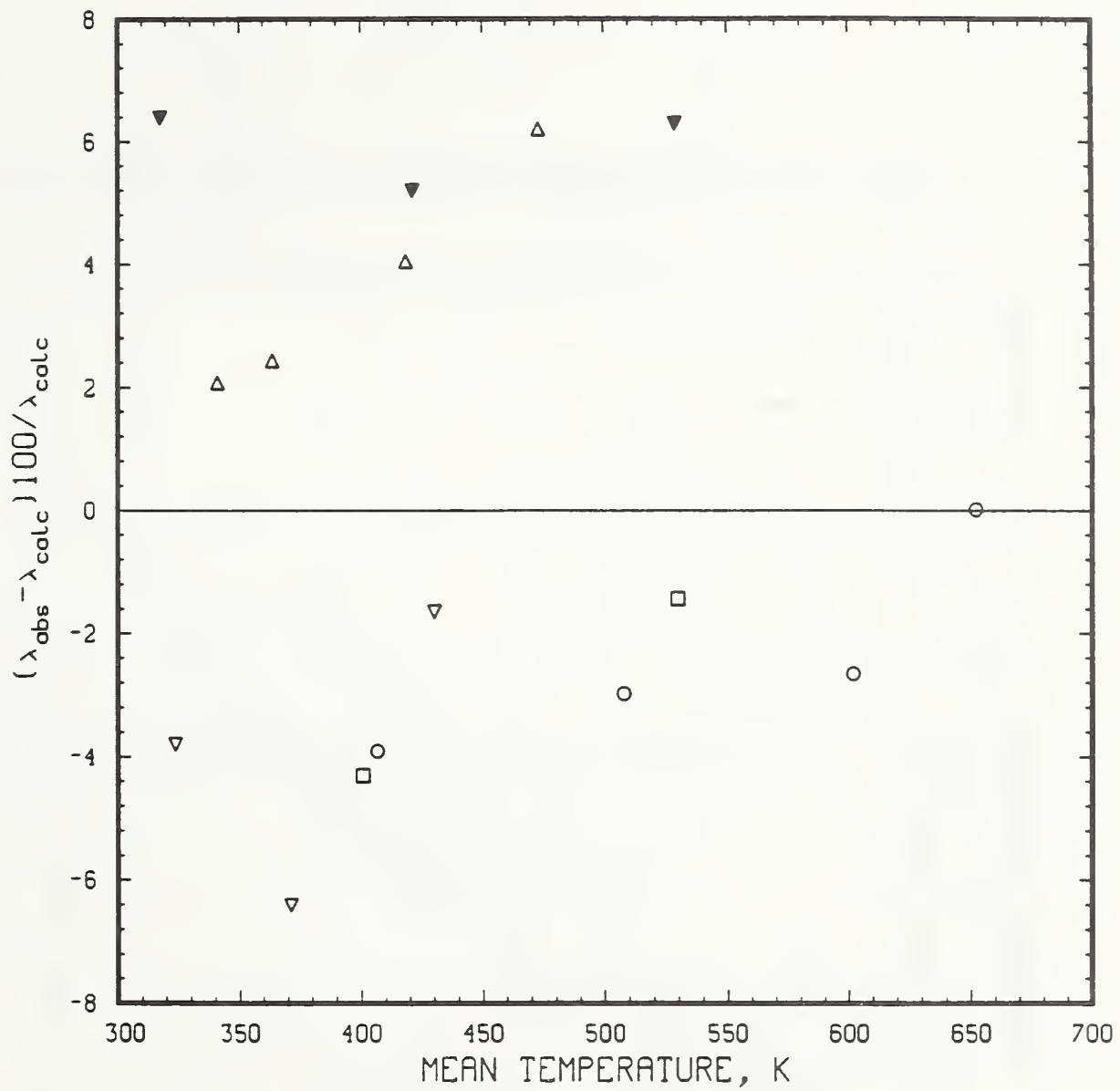points (symbols) and the fitted conductivity function.

32

Figure 6. Deviation of apparent thermal conductivity of mineral-fiber pipe
───────── insulation, as measured after conditioning, from values calcula-
ted with eq (11). Open squares represent data from laboratory 4; downward-
pointing triangles represent data from laboratory 6. Filled triangles are
repeated measurements by laboratory 6. As before, open circles represent
data of laboratory 1, and upward-pointing triangles are data of laboratory
2. Please see caption of figure 2 for an explanation of how the deviation
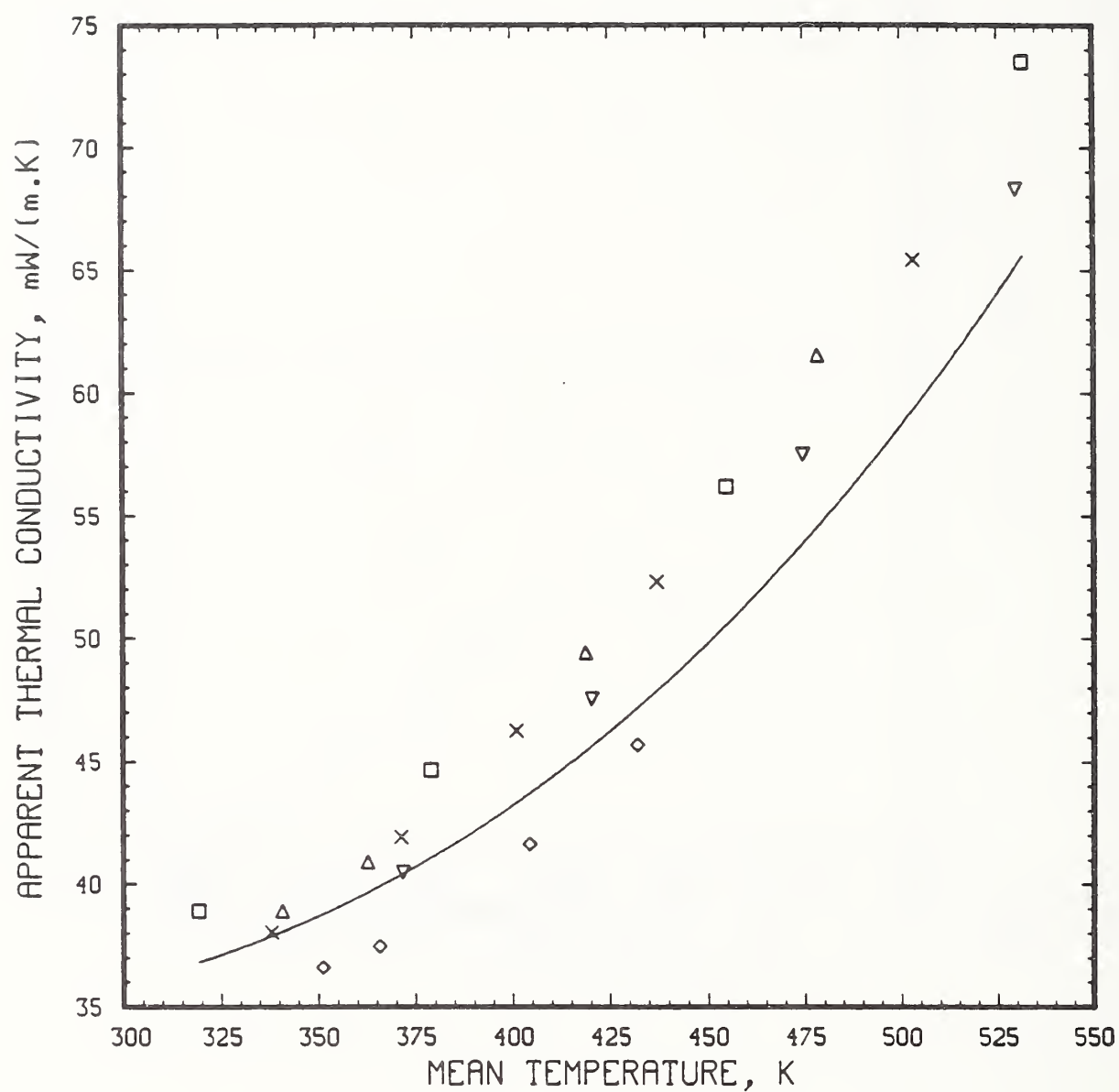is computed.

Figure 7. Apparent thermal conductivity of specimens of mineral-fiber pipe insulation that were not conditioned before they were measured. Measurements were performed in air at ambient pressure and over the range of mean temperatures from 318 to 531 K. The solid line is a plot of eq (12) for thermal conductivity as a function of temperature. Please see the caption of figure 1 for an explanation of the difference between the experimental points (symbols) and the fitted conductivity function.
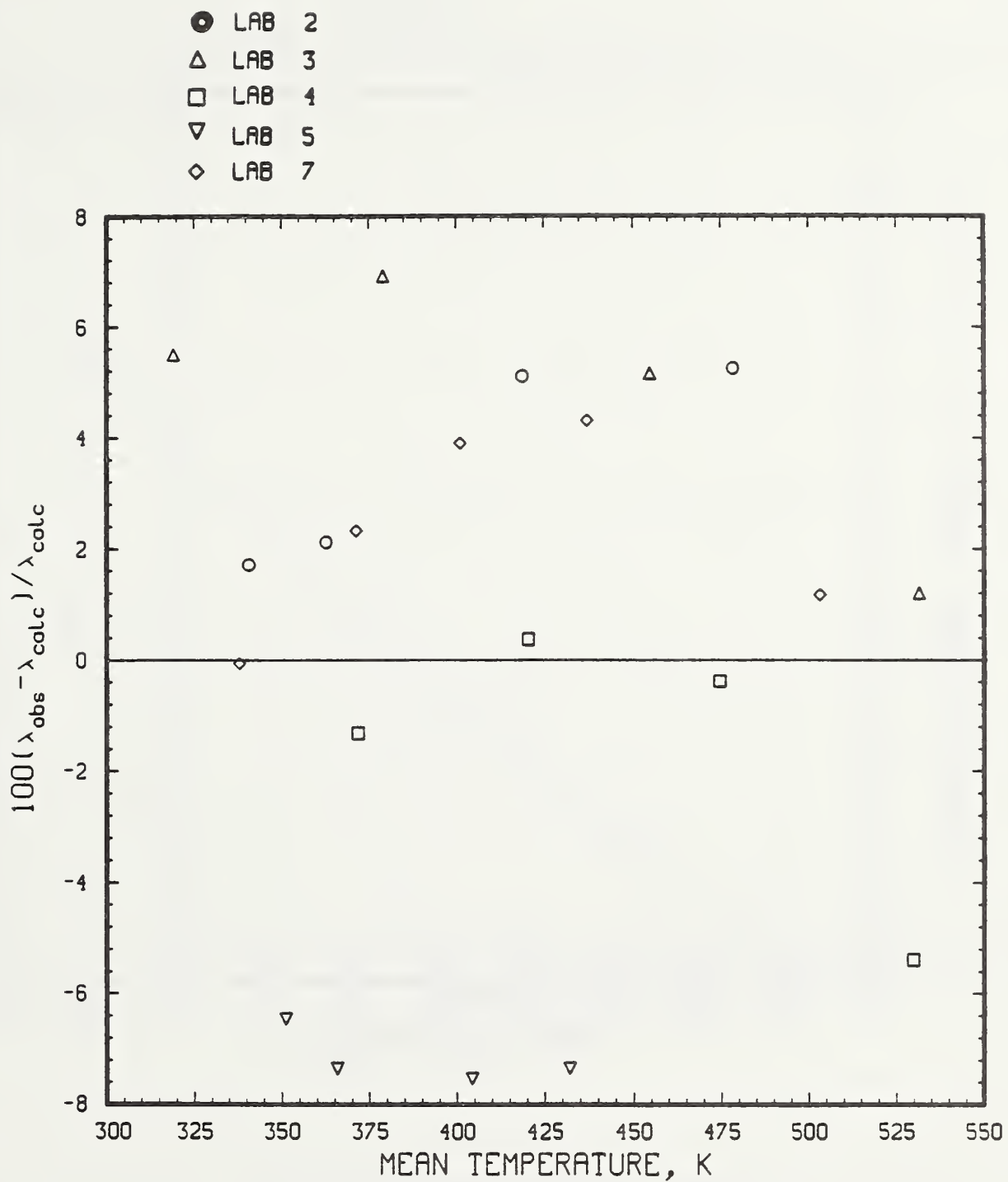
Figure 8. Deviations of apparent thermal conductivity of mineral—fiber pipe insulation, not conditioned before being measured, from values calculated with eq (12). Please see caption of figure 2 for an explanation of how the deviation is computed.
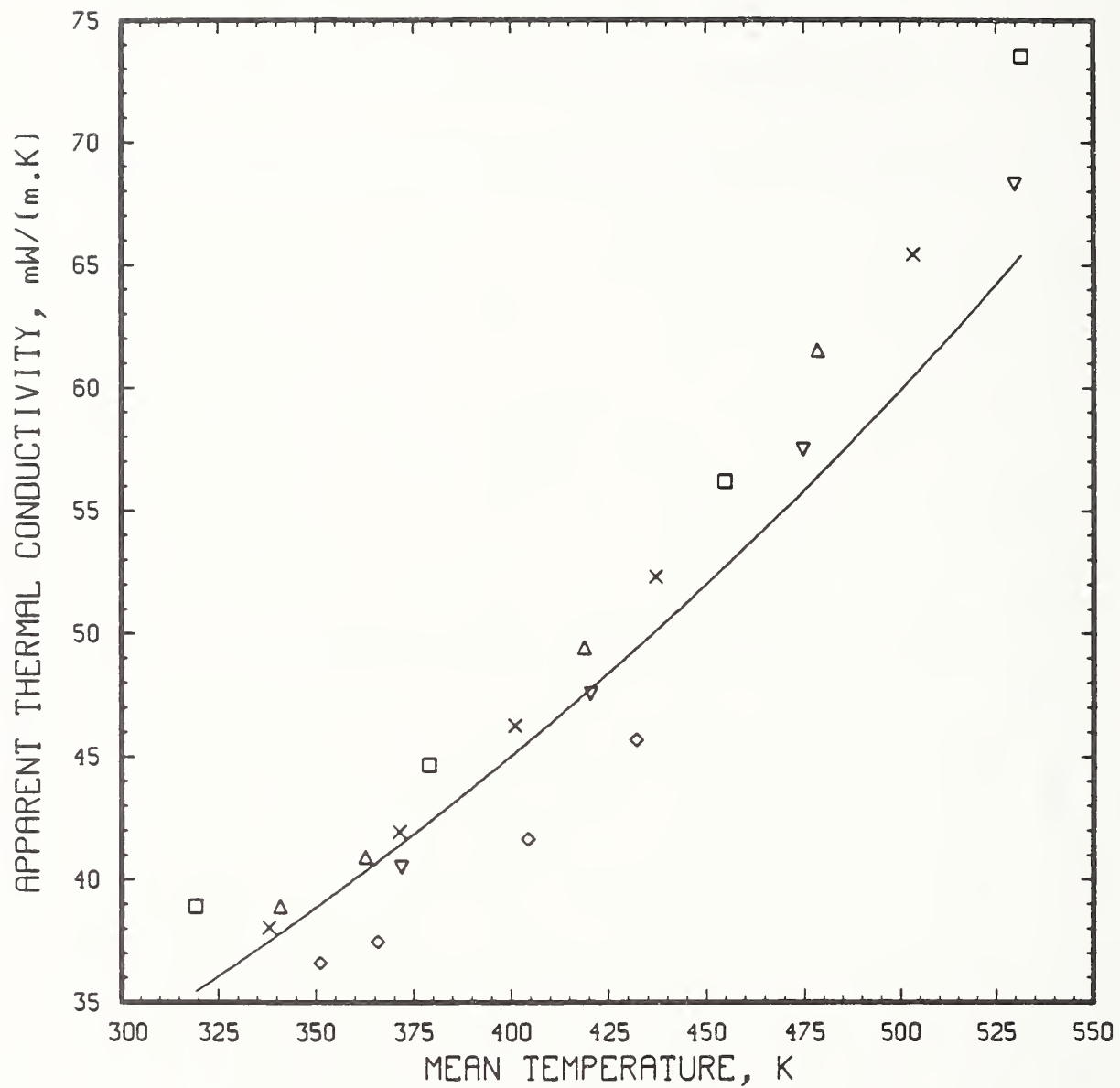
Figure 9. Apparent thermal conductivity of specimens of mineral-fiber
————— pipe insulation that were not conditioned before they were
measured. Measurements were performed in air at ambient pressure. The
The solid line is a plot of eq (11), thermal conductivity as a function
of temperature, for specimens that were conditioned before they were
measured. The position of the solid line was adjusted vertically to
minimize the rms deviation of this data from the curve. Please see the
caption of figure 1 for an explanation of the difference between the
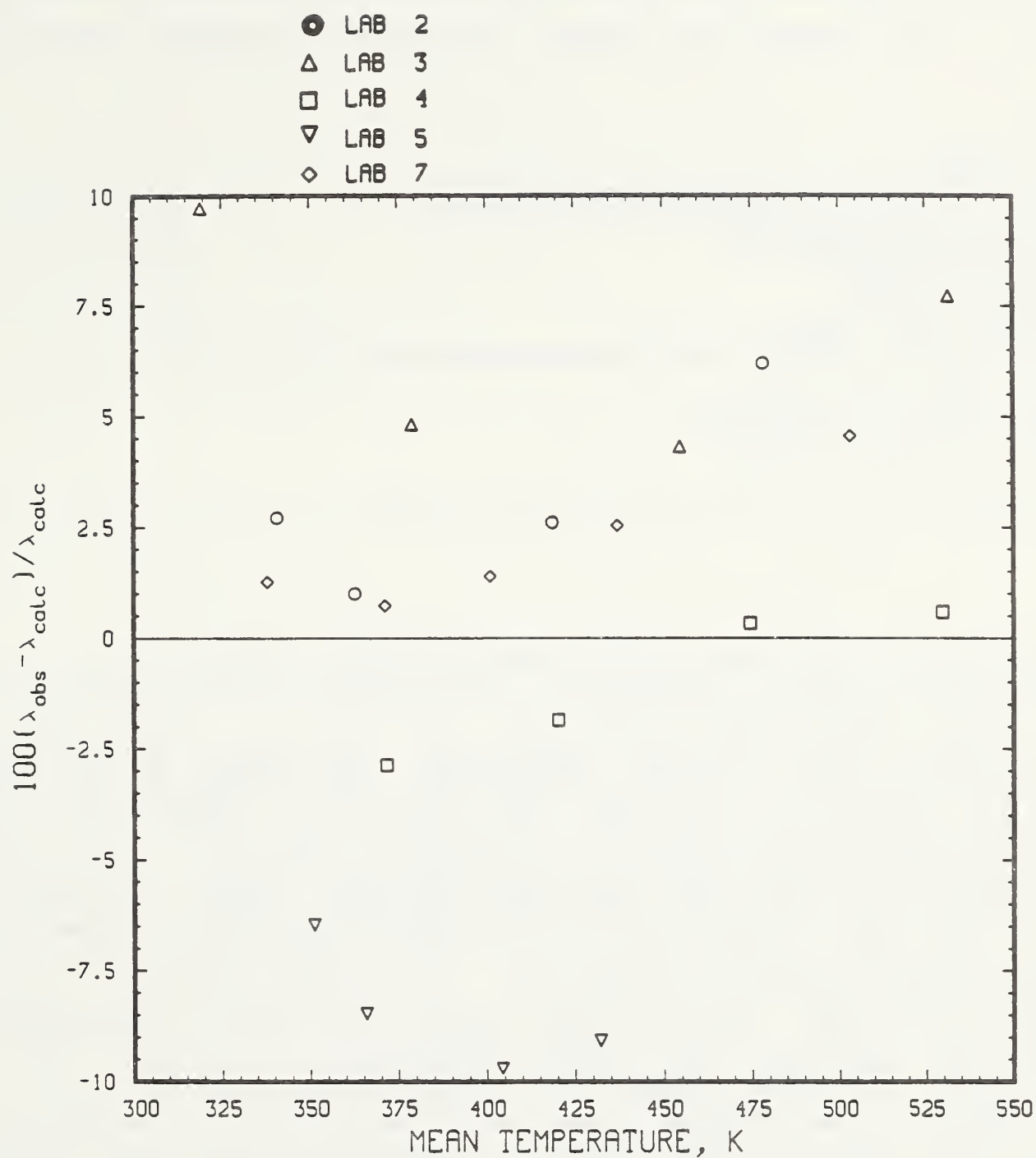experimental points (symbols) and the fitted conductivity function.

Figure 10. Deviations of apparent thermal conductivity of mineral-fiber pipe insulation, not conditioned before being measured, from values calculated with eq (11), the relation for thermal conductivity of specimens that were conditioned. Please see caption of figure 2 for an explanation of how the deviation is computed.

NBS-114A (REV. 2-80)

U.S. DEPT. OF COMM. BIBLIOGRAPHIC DATA SHEET (See instructions)	1. PUBLICATION OR REPORT NO. NISTIR 89-3913	2. Performing Organ. Report No.	3. Publication Date April 1989

4. TITLE AND SUBTITLE

INTERLABORATORY COMPARISON OF THE GUARDED HORIZONTAL PIPE-TEST APPARATUS

Precision of ASTM Standard Test Method C-335 Applied to Mineral-Fiber Pipe Insulation

5. AUTHOR(S)

David R. Smith

6. PERFORMING ORGANIZATION (If joint or other than NBS, see instructions)

National Institute of Standards and Technology
~~NATIONAL BUREAU OF STANDARDS~~
DEPARTMENT OF COMMERCE
~~WASHINGTON, D.C. 20234~~
Gaithersburg, MD 20899

7. Contract/Grant No.

8. Type of Report & Period Covered

9. SPONSORING ORGANIZATION NAME AND COMPLETE ADDRESS (Street, City, State, ZIP)

U.S. Department of Energy
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37830

10. SUPPLEMENTARY NOTES

☐ Document describes a computer program; SF-185, FIPS Software Summary, is attached.

11. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)

Apparent thermal conductivity of refractory pipe insulation from the same production lot was measured by seven different laboratories. The purpose of this intercomparison was to assess the precision and bias of the ASTM Test for Measurement of Steady-State Heat-Transfer Properties of Horizontal Pipe Insultation (C 335). Four laboratories submitted conductivity measurements for conditioned specimens. The test results for thermal conductivity of the four conditioned specimens include the temperature range from 318 to 652 K and illustrate the interlaboratory reproducibility as well as the temperature dependence of the conductivity. The standard deviation of the data from each participating laboratory was measured by their deviation from a polynomial function fitted to all data. For the 16 test results from the four laboratories that conditioned their specimens, the standard deviation was 4.3 percent. For all test results from all seven participants, the standard deviation was 5 percent. This value is offered as the estimated precision of the horizontal pipe-test method. The accuracy of this pipe-test method cannot be estimated from the data obtained in this intercomparison.

12. KEY WORDS (Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)

accuracy; apparent thermal conductivity; bias; guarded hot plate; high temperature; horizontal pipe; insulation; interlaboratory comparison; mineral fiber; precision; standard text method

13. AVAILABILITY	14. NO. OF PRINTED PAGES
☒ Unlimited ☐ For Official Distribution. Do Not Release to NTIS ☐ Order From Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.	48
☒ Order From National Technical Information Service (NTIS), Springfield, VA. 22161	15. Price